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PRODUCT SAFETY INVESTIGATION REPORT SI-4

PCS200300174 May 26, 2003

[redacted]

A SAFETY INVESTIGATION OF [redacted] (MIPKP/NMP with A.O. = 6.5%)

Project Identification

BU: 11 SPPU: PSU: Project No: Task No: R-0379-005

Drafted by Department

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Distribution

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1. OBJECTIVE

- To carry out a final product safety investigation.
- To achieve that the safety investigation results of the entitled product are documented.

2. INCENTIVE

The safety of a product is an integral part of the know-how required for its synthesis, handling, storage and transport. To detect the limits of safety, several investigations were carried out at the request of the PCLD project leader J.A.J. Reit.

3. CONCLUSIONS

3.1. According to transport recommendations the product will be accepted for transport as:
- Organic peroxide type F, liquid, UN 3109
- Packing group II
- Packing method OP 8 (max. 200 kg/225 L)

3.2. According to UN definitions the thermal stability data of the product are:
- SADT : 50°C
- Emergency temperature : 45°C
- Control temperature : 40°C

3.3. The flashpoint of the product is > 70°C.
The auto-ignition class of the product is T_3 (as a standard).

3.4. According to storage directives the product will be classified for storage as:
- Storage group 2 (CPR3)
- OP Ib (UVV)
- Type - (CS21)
- Group - (SÄIFS)

4. RECOMMENDATIONS

For transport in IBC's or tanks, venting tests have to be executed to determine the vent capacity and/or appropriate concentration for bulk.

Depending on the SADT in bulk, the product may require temperature controll.

5. GENERAL AND ANALYTICAL DATA

- Composition : [redacted]
- Molecular formula :
- Assay : %
- Act. oxygen content : 6.5%
- Physical form : Colourless liquid
- Density (apparent) : 1.15 g/ml (estimated from SADT)
- Unique code (when known) :
- Product number :
- Batch number : VRS 03021

6. FLOW-CHART DATA CLASS 5.2

6.1. Propagation of detonation

Method : UN detonation-test, A6 (Ref. 13.1)
Sample condition : [redacted]

Observations : fragmented length : 249

Result : No *(no – partial – yes)*

Exit : 1.3

6.2. Propagation of deflagration

6.2.1. Method : Time-Pressure test, UN test C1 (Ref. 13.1)
Sample condition : [redacted]

Observations : time : > 30 ms

Result : Yes, slowly *(no – yes, slowly – yes, rapidly)*

6.2.2. Method : Deflagration in a 48 mm Dewar vessel, UN test C2 (Ref. 13.1)

Conditions : emergency temperature : 45°C

Observations : deflagration rate : 0.16 mm/s

Result : No *(no – yes, slowly – yes, rapidly)*

Final result : No *(no – yes, slowly – yes, rapidly)*

Exit : 5.3

6.3. Heating under defined confinement

6.3.1 Method : Dutch Pressure Vessel test, UN test E2 (Ref. 13.1)

Observations : limiting diameter : 2 mm (with 10 g)
t_1 : 42 s
t_2 : 2 s

Result : Low *(no – low – medium – violent)*

6.3.2 Method : Koenen test *(Stahlhülsen-Verfahren)*, UN test E1 (Ref. 13.1)

Observations : limiting diameter: 1 mm
t_1 : 34 s
t_2 : 12 s
type of fragmentation : A

Result : Low *(no – low – medium – violent)*

Final result : Low *(no – low – medium – violent)*

Exit : 9.3, 11.1

6.4. Explosive power

6.4.1. Method : Modified Trauzl test, UN test F4 (Ref. 13.1)

Observations : net block expansion :3.1 ml

Result : Low *(no – low – not low)*

6.4.2. Method : High pressure autoclave, UN test F5 (Ref. 13.1)

Test not carried out.

Final result : Low *(no – low – not low)*

Exit : 12.2

7. THERMAL STABILITY

7.1. Differential Scanning Calorimeter

Test not carried out

7.2. Mini-HAST (Mini-Wärmestau)

Test not carried out.

7.3. Isothermal DSC

Test not carried out

7.4 Heat Accumulation Storage test (Wärmestau-Lagerung), UN test H4 (Ref. 13.1)

Conditions : 0.5 l cylindrical Dewar vessel
half-life time of cooling with 400 ml DMP : 10.0 hrs
intake of 400 ml sample : 460 g

Observations : self-accelerating decomposition at : 50°C
induction period at °C : 3 days
type of decomposition : mild *(violent – mild)*
temperature with no self-acceleration : 45°C
maximum temperature rise at °C : 1°C
relative loss of active oxygen at °C : - %

Result : SADT is 50°C, see figure 2 and 3

According to UN definitions the expected *(listed – expected)* values are :
SADT : 50°C
Emergency temperature : 45°C
Control temperature : 40°C
Expected storage temperature : 25°C (max.)

8. SDS DATA

8.1. Flashpoint

Method : Setaflash (ISO 3679), EU test A9 (Ref. 13.2)

Result : Flashpoint is > 70°C

8.2. Auto-ignition temperature (exploratory test)

Reported results from earlier tests : Yes *(no – yes)*
Reference SI report : PCS 200100482

Method : DIN 51794, EU test 15 (Ref. 13.2), only screening

Observation : 300°C

Result : Temperature class is T_3 (as a standard (Ref. 13.3))

9. BURNING PROPERTIES

9.1. Ignitability

Test not carried out

9.2. Burning test (storage)

Method : laboratory-scale fire test (Ref. 13.4, 13.5 and 13.6)

Conditions : temperature : 25°C

Observation : burning rate : 2.5 kg/m^2.min

Result : according to CPR-3 : storage group 2
according to UVV : OP Ib
according to CS-21 : -
according to SÄIFS : -

10. MISCELLANEOUS

10.2.2. Impact-hammer test

Reported results from earlier tests : Yes *(no – yes)*
Reference SI report : PCS 200100482

Method : BAM-method, UN test 3 (a) (ii) (Ref.13.1) and EC test A14, part impact Sensitivity (Ref. 13.2)

Observations : at 40 J : positive *(negative – positive)*
at 7.5 J : negative *(negative – positive)*
at 2 J : negative *(negative – positive)*

Result : Impact sensitivity is Medium *(low – medium – high – very high)*

10.5. Sensitivity to static charge (non-polar material only)

Method : BS 5958, see PCS test method C-SR-044 (Ref. 13.7)

Condition : temperature : 20°C

Observation : 62.10^6 pS/m

Result : Sensitivity to static charge is Low *(low – medium – high)*

10.6 Behaviour at low temperatures

Reported results from earlier tests : Yes *(no – yes)*
Reference SI report : PCS200100482

10.6.2. Method : Cold storage test

Conditions : temperature : -20°C
tube : 28 mm (ID)
duration : 7 days

Observations : solidification : No *(no – yes)*
crystallisation : No *(no – yes)*
phase separation : No *(no – yes)*

phenomenon reversible : - *(no – yes)*
at : -°C

Result : Increase in hazard due to physical changes at low temperatures : No *(no – yes)*

10.6.3 Additional tests

Crystallisation tests at one litre scale have been executed with the following samples:

1) VRS 03001, T4/T3 = 4.55 (i.e. sample as tested in this SI-4)

2) VRS 03002, T4/T3 = 3.58 (i.e. aged sample; 8 weeks 30 °C, see PCS 200300236 for product safety check)

Tests were executed at temperatures of –15 and –25 °C for 3 months. No crystallisation was observed.

10.9 Gas evolution test

10.9.1. first test

Conditions : temperature : 25°C

Observations : Gas Evolution Rate : ≤ 0.4 ml gas per 100 ml product per day

10.9.2. second test

Conditions : temperature : 25°C

Observations : Gas Evolution Rate : ≤ 0.4 ml gas per 100 ml product per day

Result : Vent cap required : No *(no – yes)*

11. DISCUSSION

The results of a safety investigation of aged product has been reported in a separate safety-detail report (Ref. 13.10).

12. PRECEDING PRODUCT SAFETY INVESTIGATION FORMS

- SI-1 : date : ; batch number :
- SI-2 : date : ; batch number :
- SI-3 : date : 2000-06-27 ; batch number : VRS 00071

13. REFERENCES

13.1. Current United Nations, Recommendations on the transport of Dangerous Goods, manual of tests and criteria.
13.2. Annex V, EU-directive 92/69/EU
13.3. “AIT T3 as a standard”
13.4. CPR-3, Storage of Organic Peroxides
13.5. UVV BGV B4, Organische Peroxide
13.6. SÄIFS 1996:4, Organiska Peroxider
13.7. ACRD 950-800, Method for the measurement of the electrical conductivity, by P. Schuurman, dated 13-09-1995
13.8. ACRD 970-0368, Method for the measurement of vapor pressure with DSC 27HP, by G.B. Boerdijk, dated April 10, 1997
13.9. Tests and criteria of the safety investigations SI-1 till SI-4, by Cluster Product Safety. To be published soon.
13.10 PCS 200300236, A product safety check of aged TC-R-3005 (8 weeks at 30°C).

14. APPENDICES

[redacted]

Figure 2

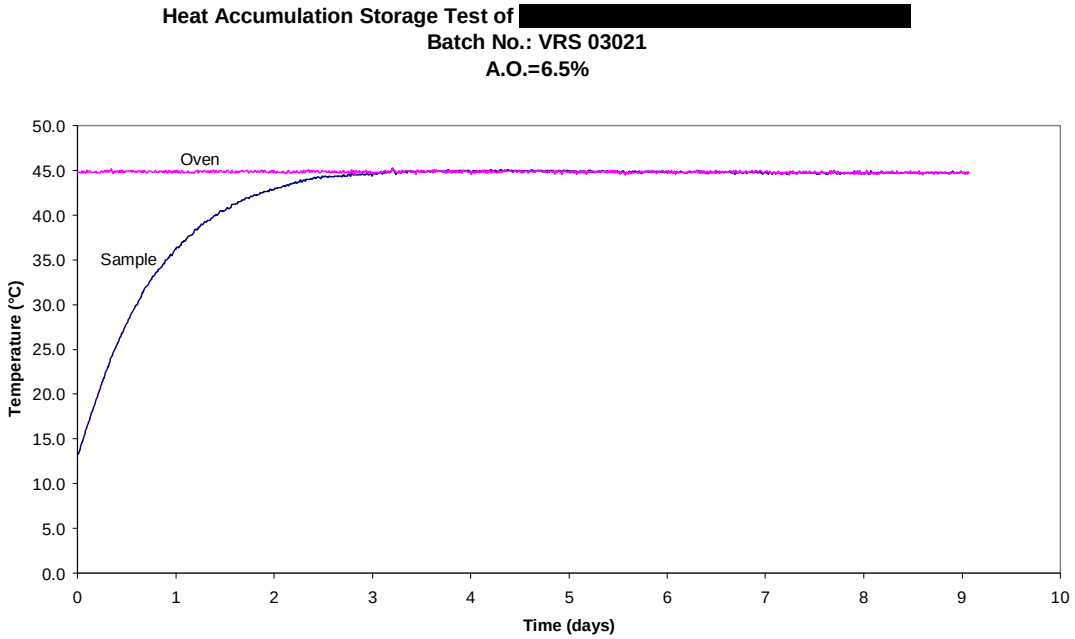

Figure 3